Name:_____

Introduction to Analytical Chemistry
Chem. 210 Sections 001 and 002
Exam II

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Spring 2005

Answer all the questions. **DO NOT** write on this examination paper, use the blank sheets at the end of the exam for your answers. Credit will not be given for numerical problems unless all relevant calculations are shown. Please give answers to numerical questions to 3 significant figures unless asked otherwise.

1. a) The red pigment in a fourteenth century manuscript was analyzed for its lead content with the following results:

Determination	Pbppm
1	10
2	12
3	24
4	9
5	10

i) For the data set calculate the confidence interval at the 95% confidence level.

15 points

ii) The authenticity of the manuscript is in doubt. Manuscripts from this era typically have lead concentrations of 13ppm in their red pigments. You are asked to give your opinion on the authenticity of the manuscript based on your statistical analysis of the data. What conclusion would you draw? In addition, justify that conclusion?

15 points

2. a) List the general steps that you would perform in a gravimetric analysis.

10 points

b) List the properties that are needed in a precipitation reagent.

5 points

c)

i) What is the approximate minimum amount of analyte that needs to be present in a sample for a gravimetric analysis?

2 points

ii) What is the approximate accuracy of a gravimetric analysis?

2 points

3. a) Solve the following arithmetic problem:

$$\frac{13.721 \pm 0.006 - 9.781 \pm 0.003}{22.170 = 0.004} = ?$$

i) What is the absolute error in the answer?

ii) Give the answer to the correct number of significant figures.

10 points

b) A solution of sodium sulfide and a solution of copper (II) nitrate are mixed together such that the sodium sulfide is in a stoichiometric excess Copper (II) sulfide precipitates from the solution as a colloidal precipitate. Draw a diagram and give an explanation of a colloidal particle in the solution showing its inner and counter ion layer with the appropriate ionic species in each layer.

c) Define or explain the following:

i) Coagulation

ii) Digestion

iii) Isomorphic Inclusion

iv) Non-Stoichiometric compound

4 points

d) What are the structural characteristics needed for an organic precipitating agent that forms a complex with a metal cation?

2 points

4. a) 0.400g of copper(II) sulfate is dissolved in water. The copper is precipitated as copper (II) iodate ($Cu(IO_3)_2$). What is the mass of copper (II) iodate precipitated.

12 points

b) The aluminum in a 1.200g sample of impure ammonium aluminum sulfate was precipitated with aqueous ammonia as the hydrous $Al_2O_3 \cdot xH_2O$. The precipitate was filtered and ignited at 1000^oC to give anhydrous Al_2O_3 which weighed 0.1798g. Calculate the per centage of aluminum in the sample.

13 points

ANSWERS

1. Perform a Q test on determination '3'.

   i) $\mu = 10 \pm 2$ ppm Pb

   ii) Null Hypothesis: 'There is no significant difference between the red pigments in the manuscript and authentic fourteenth century manuscript.

$$\mu - \bar{x} = 3 \text{ ppm Pb}$$

$$\frac{ts}{\sqrt{N}} = \pm 2$$

   at 95% level $\mu - \bar{x} > \pm \frac{ts}{\sqrt{N}}$.

   At the 95% confidence level we would conclude that the null by pothesis fails and a significant difference exists between the red pigments. At this confidence level we would conclude that 95 times out of a 100 we would be correct in concluding that the manuscript is not authentic.

   If we had listed at the 99% confidence level we would have found:

$$\mu - \bar{x} = 3 \text{ ppm Pb}$$

$$\frac{ts}{\sqrt{N}} = 3.7$$

   at 99% level $\mu - \bar{x} < \pm \frac{ts}{\sqrt{N}}$.

   At the 99% confidence level we would conclude that we cannot tell any difference between the red pigments. Therefore, if we wish to be 99% sure the best we can say is that we cannot tell any difference between the red pigments. This does not mean that there is no difference only that we cannot tell any difference.

2. a)
   i) Accurately weigh a sample to be analyzed.
   ii) Dissolve the sample
   iii) Remove any interferences
   iv) Adjust the experimental environment for the best results
   v) Add a precipitating reagent
   vi) Precipitate in hot solution
   vii) Filter the precipitate
   viii) Wash the precipitate
   ix) Dry the precipitate
   x) Calculate the concentration of analyte in the sample

b)

i) Solubility: - need a precipitate with as low a solubilities as possible
ii) Ease of filtering
iii) Stoichimetry: - must have a reproducible stoichimetry
iv) High molecular weight
v) Non hydroscopic
vi) Selectivity: – Ideally needs to be specific for the analyte – not possible

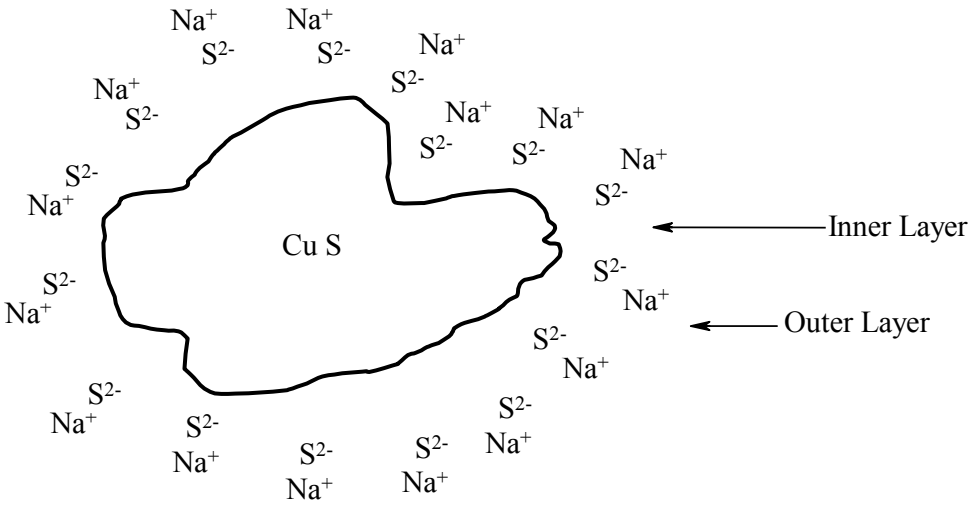
c) ~ 1-5 %

d) ~ 3ppt

3. a)

i) $S_{abs} = \pm 0.0003$
ii) 0.1771

b)



As the anion of the colloidal particle is present in excess in solution then that ion will be absorbed on to the colloidal particle forming the inner layer, in this case S^{2-}. The counter ion layer or outer layer must contain a cation in this case sodium ion is a reasonable choice.

c)

i) Coagulation: – This is the process by which colloidal particles come together to form larger particles.

ii) This is the process of gently heating a solution containing a crystalline precipitate in order to remove impurities from the precipitate.

iii) This is a process by which an anion or cation in a crystal lattice is replaced by a similar but different cation or anion. The result is an impure crystal.

iv) These are compounds which do not have a fixed reproducable stoichiometry.

d)

i) These are complex organic compounds.

ii) They have at least two functional groups.

iii) The functional groups have at least one lone pair of electrons.

4. a) 1.04 g

b) 7.30%